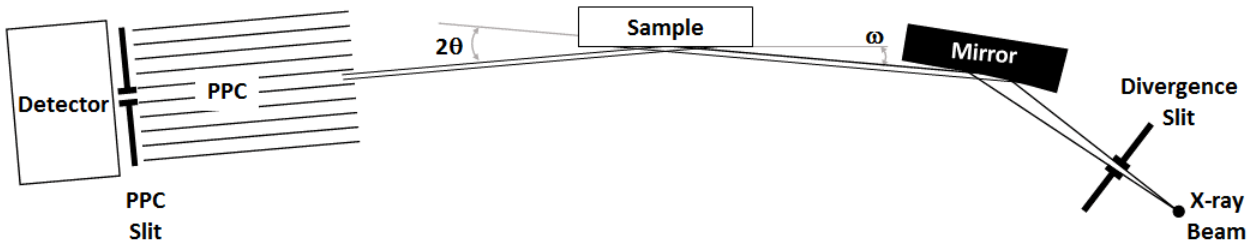
Reflectivity



I. Login

1. *Enable* instrument in **Badger**.
2. Start **Data Collector**.
3. Type your "User Name" and "Password".
4. Select *Instrument* → *Connect*.
5. Choose Configuration **Mirror + PPC**.
6. Click *OK*.

II. Hardware Setup

1. X-ray Tube is in "Line Focus".
2. Goniometer Resolution set to "Normal 0.001 deg".
3. Incident Beam Optics – **Mirror**
   Note: if you have to change incident beam optics please first turn Automatic attenuator to "Activate" status and then unplug attenuator cable.
   a. Insert 1/32° **Divergence Slit** into **Mirror** optics.
   b. If sample's vertical dimension is smaller than 25 mm, insert correct size **Mask**.
4. Diffracted Beam Optics – **Parallel Plate Collimator 0.27°**
   a. Insert **PPC Receiving Slit** into **Parallel Plate Collimator** optics.

III. Data Collector Software

1. Select the **Incident Beam Optics** tab.
   a. Double click any item. **Incident Beam Optics** window will appear.
   b. Go through all tabs and select proper optic components:
      - *PreFIX Module* – select **Mirror**.
      - *Divergence Slit* – select 1/32° **Divergence Slit**.
      - *Anti-Scatter Slit* – select **None**.
      - *Mask* – select appropriate **Mask**.
      - *Beam Attenuator* – **Progr. Beam Attenuator**. For initial alignment set *Usage* = "Do not switch" and *Status* = "Activated". Make sure the *Description* = "Mirror". If not, click *Select* and select Mirror attenuator.
      - *Filter* – select **None** or **Beta filter** if you will be using one.
2. Select the **Diffracted Beam Optics** tab.
   a. Double click any item. **Diffracted beam optics** window will appear.
   b. Go through all tabs and select proper optic components:
      - *PreFIX Module* – select **Parallel Plate Collimator 0.27°**.
      - *Anti-Scatter Slit* – select **None**.
      - *Receiving Slit* – selsct **PPC Receiving Slit**.
      - *Filter* – select **None**.
      - *Monochromator* – select **None** or **Flat Graphite Monochromator** if you use one. *By default there is no monochromator mounted.*
3. Select **Instrument Settings** tab.
   a. Double click any item in the tree view to prompt another window.
   b. Press **X-ray** tab. Set generator power to 45 kV and 40 mA.

IV. Diffractometer Zero Alignment

1. In **Instruments Settings** check **Z** position. If it is larger than 5mm move it back to at least 5mm.
2. Move all other motors to zero positions.
3. From Menu select *Measure* → *Manual Scan*.
4. From the *Scan Axis* drop down menu select **2Theta**.
5. Enter *Range* = 1°, *Step Size* = 0.005°, and *Time per Step* = 0.2sec. Then press *Start*.
6. After scan is finished, move **2Theta** axis to a peak position using one of the two ways:
   a. Peak Mode. Right click on mouse and select *Peak Mode*. New window will appear showing the **2Theta** position of the peak. Click *Move To*. Close the window.
   b. Move Mode. Right click on mouse and select *Move Mode*. Move **2Theta** to the center of the mass of the peak.
7. Select *User Settings* → *Sample Offsets* and set current **2Theta** position to zero.
8. Note the direct beam intensity.

V. Sample Mounting

1. Mount sample using scotch tape. Long direction should be horizontal. If the sample is large, supplied clips can be used to hold the sample.
2. If in the **Instrument Settings** tab **X** = 0.0 and **Y** = 0.0, beam is positioned at the center of a sample stage (aluminum disk).

VI. Moving Sample into the Beam Position Using Direct Beam

1. Note the direct beam intensity. In **Instruments Settings** move **Z** to higher values until intensity starts to drop.
2. **Z** alignment can be performed using either optimization program or manually:
   a. Using optimization program.
      - Select *Measure* → *Program*. New window with user written programs will appear.
      - From the *Measurement Type* select *Optimize Program*.
      - Find proper program that says "Opt Z" and select it.
      - Click *OK* and start the scan.
   b. Manually.
      - *Select Measure* → *Manual Scan*.
      - In **Manual Scan** window from the *Scan Axis* drop down menu select **Z**.
      - *Enter Range* = 2mm, *Step Size* = 0.01mm, and *Time per Step* = 0.2sec. Press *Start*.
      - *After* scan is finished, right click on mouse and select *Move Mode*.
      - Move **Z** to the intensity value corresponding to ½ of the direct beam intensity.

VII. Aligning Sample Parallel to the Beam

1. In **Manual Scan** window from the *Scan Axis* drop down menu select **Omega**. Enter *Range* = 2°, *Step Size* = 0.01°, and *Time per Step* = 0.2sec. Then click *Start*.
2. After scan is finished, right click on mouse and select *Move Mode*. Move *Scan Axis* to the center of gravity of the peak.
3. Realign Z using one of the methods in part **VI**.
4. Repeat steps 1-3. If after **Omega** scan peak center is positioned at the hairline location, alignment is complete.
5. In *User Settings* → *Sample Offsets* set current **Omega** position to zero.
6. In the **Incident Optics** tab set *Automatic Attenuator Usage* = "Preset Intensity" with *Activate Level* = 500,000 and *Deactivate Level* = 450,000.

VIII. Optimizing Reflected Intensity

1. In **Instrument Settings** tab enter **2Theta** = 2°, **Omega** = 1°. Click *OK*. Diffractometer will move to **2Theta** = 2°.
2. In the **Manual Scan** window set *Scan Axis* **2Theta-Omega**. Enter *Range* = 4°, *Step Size* = 0.02°, and *Time per Step* = 0.2sec. Then click *Start*.
3. In the measurement window right click on mouse and select *Axes* → *Logarithmic Scale*.
4. After scan is finished, right click on mouse and select *Move Mode*. Move *Scan Axis* to a position just to the right of the critical angle or to the maximum intensity of the first

(lowest angle) clearly visible thickness fringe.

5. In the *Manual Scan* window set *Scan Axis* **Omega** Enter *Range* = 0.5º, *Step Size* = 0.002º, and *Time per Step* = 0.2sec. Then click *Start*.
6. After scan is finished select *Axes* → *Linear Scale*. Right click on mouse and select *Peak Mode* or *Move Mode*. Move *Scan Axis* to the center of gravity of the peak.
   Note 1: *If initial and final* ***Omega*** *positions differ by more than 0.1º, it might be necessary to readjust* ***Z*** *position as described in part VI.*
   Note 2: *If two broad low intensity peaks are present, reflectivity on this sample will not be possible due to high interface roughness. If three peaks are present, with a sharp peak in the middle, move Scan Axis to the position of the sharp peak in the middle.*
7. In the *Manual Scan* window set *Scan Axis* **Chi** Enter *Range* = 6º, *Step Size* = 0.03º, and *Time per Step* = 0.2sec. Then click *Start*.
8. After scan is finished, right click on mouse and select *Move Mode*. Move *Scan Axis* to the center of gravity of the peak.
9. After the measurement is completed press the right mouse button and select the *Move Mode*. Move *Scan Axis* to the center of gravity of the peak.
10. If necessary, repeat steps 5 and 6.
11. In *User Settings – Sample Offsets* set current **Omega** position to ½ of **2Theta** position.

IX. Measurement

1. In the **Incident Beam Optics** tab set *Beam Attenuator Usage* – "Preset Intensity" with *Activate Level* = 500,000 and *Deactivate Level* = 450,000.
2. Simplest way to execute scan is to do a **Manual Scan**. It is a relative scan i.e. executed around current goniometer position with the range specified in **Manual Scan** window.
3. To do *2Theta-Omega* scan first move **2Theta-Omega** to the middle position of the scan range.
4. In **Manual Scan** window select *Scan Axis* **2Theta-Omega** and appropriate *Range*, *Step Size* and *Time per Step*. Click *Start*.
5. When scan is completed, save it through *File* → *Save As* menu.
   Note 1: *Manual Scan will be lost if it is not saved.*
   Note 2: *Reflectivity data is best viewed in Logarithmic Scale.*

X. Logging out

1. Close the shutter.
2. Beam *Attenuator – Usage* = "Do Not Switch" and *Status* = "Activated".
3. Move all angles to zero positions and **Z** to 5 mm.
4. Lower the power of the x-ray tube to 40 kV and 20 mA.
5. Close **Data Collector**.
6. *Disable* instrument in **Badger**.